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Torrefied biomass filled polyamide biocomposites: mechanical and physical property analysis

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Abstract Biobased fillers in thermoplastics have seen increased usage over the last several years. The increased usage of biobased fillers follows the ever-increasing thrust to reduce petroleum and synthetic petrochemical product consumption. The study of polyolefin-based biocomposites is well established, however, the incorporation of biomass fillers into engineering thermoplastics still proves to be challenging. For engineering thermoplastics, the increased processing temperatures lead to degradation of the biomass, often detrimental for the mechanical performance and visual appearance of the end product. The goal of this work was to evaluate the effectiveness of agricultural by-products as fillers in polyamides, while minimizing the effects of increased processing temperatures. Through the use of torrefaction, polyamide biocomposites were produced and shown to have comparable mechanical properties to the neat matrix. Torrefied sunflower hulls and flax shive were shown to produce tensile strengths within 70 % of the neat matrix, increase elastic modulus by 150 %, maintain flexural strengths within 94 % of the neat matrix, increase flexural modulus by 154 %, and decrease the absorption of environmental moisture by as much as 50 %.

Introduction

Over the last decade, the replacement of traditional synthetic fillers by biobased fillers in thermoplastics has seen a remarkable increase. The increasing push to reduce petroleum dependence along with the low cost to density ratio have aided the increased acceptance of biobased fillers [1–6]. For the automotive industry, the fastest growing consumer of polyamides, the push to reduce petroleum usage has led to more widespread use of biocomposites for interior components and the replacement of heavy metal components under the hood with plastic parts [7, 8]. While the study of commodity polyolefin biocomposites is well established providing suitable replacements for interior automotive components; the research of biobased fillers in engineering thermoplastics for applications under the hood is quite sparse. By providing a biocomposite that can withstand the processing and working temperatures of engineering thermoplastics, such as polyamides, the automotive industry could further reduce its consumption of petroleum rich plastic by as much as 30 % by weight.

As processing and working temperatures exceed 200 °C, the thermal stability of biobased fillers becomes critical to maintain mechanical integrity of the composite. Without any pretreatment the increased processing temperatures of engineering thermoplastics can lead to the degradation of fillers, compromising the mechanical integrity of the biocomposite. The densification process commonly referred to as torrefaction has been identified as an effective means of

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preparing biomass derived fillers for high temperature processing conditions.

Torrefaction, traditionally used to produce alternative energy sources, is a decomposition and densification process that converts low weight energy-light constituents within biomass into various by-products. Three distinct phases are created during the torrefaction process: a solid carbonized mass, an acidic liquid phase, and syngas. The solid carbonized mass has higher hydrophobicity than untreated biomass. Although torrefaction of biomass has been studied extensively over the last several years, the exact chemical reactions occurring during the process are still unclear. It has been shown that the breakdown of hydroxyl groups on cellulose microfibrils is the cause of increased hydrophobicity [9–15].

Natural fibers begin to significantly degrade at 140 °C when exposure times are lengthy. Torrefaction is traditionally completed in the range of 225–300 °C in an inert atmosphere for several hours. The length and temperature chosen for the process will determine the degree of torrefaction of the fibers. During the torrefaction process, hemicellulose, fats, waxes, and other low degradation point constituents within the fibers are converted yielding a biomass consisting of mostly crystalline cellulose, degrading between 300 and 375 °C, and lignin, degrading slowly over 250–500 °C [14, 15]. Torrefaction will degrade the amorphous cellulose and begin to mildly degrade the crystalline cellulose.

The solid by-product of torrefaction has the potential to be used in biocomposite production with high temperature thermoplastics such as polyamide. The addition of a more hydrophobic torrefied biomass to polyamides hinders the absorption of moisture, allowing end use parts to maintain their mechanical integrity longer than an unfilled polyamide. The syngas produced during torrefaction can potentially be burned to power the next torrefaction process making it self-sustaining after the initial torrefaction run. This work was focused on evaluating the effectiveness of torrefied sunflower hulls (TSFH) and torrefied flax shive (TFS) as reinforcement in polyamide-6 biocomposites. Mechanical performance comparable to the unfilled matrix was successfully achieved using torrefaction.

To further increase the green nature of the biocomposites, locally available agricultural by-products were chosen for the natural fibers. As one of the leading producers of sunflowers in the country, North Dakota has a natural waste stream from commodity processors in the area. The hulls or shells of the seeds removed during the commodity processing have very low nutritional value; hulls can be substituted at no more than 20 % of the feed for livestock [16]. As the demand of hulls for the purpose of livestock feed is low, the waste stream is abundant and inexpensive. On average the North Dakota region produces 1.1 million metric tons of sunflower seeds a year. With approximately 30 % of the seed mass being the hull, there is an average supply of 330,000 metric tons of sunflower hulls each year [17, 18].

The second biomass chosen for this work is flax shive. Flax shive, unlike the outer protective nature of the sunflower hull, comes from the central woody core of flax stalk. Flax straw left on the field after flax seeds are harvested goes through a decortication process to remove flax fiber, the outer most layer of the flax straw. On average each year the flax producers near North Dakota produce 1 million metric tons of flax straw. Shive makes up approximately 70 wt% of the flax straw, for an average of 700,000 metric tons of flax shive available each year [19, 20].

Materials and methods

Materials

Due to their abundant usage in commercial applications, polyamide 6 (PA6) was used for this work. The PA6 was obtained from PolyOne, Avon Lake, Ohio. Ultramid 8202 manufactured by BASF Corporation, a low viscosity general purpose homopolymer was chosen for the PA6. Table 1 shows the general properties of the PA6. Sunflower hulls were obtained from Red River Commodities, Fargo, North Dakota. The flax shive used was obtained from Flax Stalk Natural Fiber Solutions a subsidiary of Schweitzer-Mauduit International, Winkler, Manitoba, Canada.

Wet chemical analysis was performed by the Animal Sciences Department at North Dakota State University to determine the constituent makeup of the sunflower hulls and flax shive. The AOAC standard 930.15 was used in dry matter determination, AOAC standard 920.39 was used to determine crude fat, and AOAC standard 2001.11 was used for the determination of crude protein. The USDA Agricultural Handbook No. 379 was followed for the analysis of neutral detergent fiber, acid detergent fiber, and acid detergent lignin. The constituent breakdown of the fibers used in this work can be seen in Table 2.

Torrefaction

As-received biomass was torrefied using a Lucifer model 7021-GT-E high temperature convection furnace. The size of the chamber limited the amount of biomass that could be torrefied at one time to approximately 200 g. To prevent combustion of the biomass during the heating process, an inert atmosphere was maintained using argon gas at a flow rate of approximately 2,000 sccm. The biomass was heated to 300 °C and held at temperature for 8 h. The biomass was

Table 1 Material properties for the PA6 used in this work

	Melting temperature (°C)	Density (g/cm^3)	Elastic modulus (GPa)	Tensile strength (MPa)	Flexural modulus (GPa)	Flexural strength (MPa)	Impact toughness (J/m)
PA6	464–545	1.13	2.7	79.0	2.8	108.0	58.0

Table 2 Constituent breakdown for the biomasses used in this study, all numbers are weight percentage

Biomass	Lignin	Cellulose	Hemi cellulose	Moisture	Ash	Starch	Calcium	Phosphorus	Crude Fat	Crude Protein
Sunflower hull	22.4	39.8	15.1	7.1	2.7	0.6	0.3	0.17	7.0	5.3
Flax shive	21.1	40.2	16.8	5.2	2.8	0.7	0.2	0.01	0.3	2.3

then allowed to cool to room temperature in the inert atmosphere. No fractionation of the TFS was performed prior to biocomposite production. TSFH were fractionated using a blender prior to biocomposite processing. Thermogravimetric analysis (TGA) of the untorrefied and torrefied biomasses was conducted using a Texas Instruments Q500 TGA. A temperature ramp of 10 °C per minute from room temperature to 400 °C was used to analyze the biomass.

Composite production

The torrefied biomass and polymer matrices were melt compounded using a Leistritz Micro-18/GL-40D, co-rotating twin-screw extruder. All materials were dried overnight at 80 °C in a convection oven prior to extrusion. Dry blending of the matrix and fillers was completed before melt compounding based on weight percentages. Three varying weight percentages were used for each biomass, 10, 20, and 30 wt%. A temperature profile of: 193, 210, 216, 227, 238, 232, 227 °C starting at the feeding zone and ending with the metering zone was used for extrusion. The extruded biocomposites were water cooled, pelletized, and dried overnight at 80 °C in a convention oven prior to injection molding. Test specimens with a thickness of 3.2 mm were molded using a Technoplas, Inc. Model Sim-5080 injection molder. All molding temperatures were kept below 300 °C.

Mechanical characterization

Tensile strength and elastic modulus were determined according to ASTM standard D638 using an Instron Model 5567 load frame. Five specimens of each biocomposite grade were tested at a rate of 5 mm/min. The same Instron load frame was used to determine flexural strength and modulus according to ASTM standard D790 using five specimens. Specimens were tested at an average rate of 1.4 mm/min. Notched Izod Impact toughness was determined according to ASTM standard D256 using six specimens for each grade. Specimens were notched with a Veekay Testlab Veekay Notch Cutter. A Tinius Olson Model IT 504 Impact 104 with a pendulum weight of 4.497 N was used for impact testing. Moisture uptake was analyzed using an Arizona Instruments Computrac 4000XL Moisture Analyzer. Samples were soaked for 24, 72, and 168 h in distilled water, once removed from the water any adsorbed surface water was removed by towel drying prior to analysis. The immersion density of the biocomposites was determined using a Mettler Toledo 33360 immersion density kit, the observed densities were then used to back calculate the density of the torrefied biomass.

Results and discussion

In order to evaluate the effectiveness of torrefied biomass as filler in polymer matrices, a control experiment of untreated and TSFH was conducted prior to full experimental matrix testing. Figure 1 shows the tensile properties measured for the untreated and TSFH. The TSFH showed a 54 % increase in tensile strength over the non-TSFH. The strength advantage is believed to come from a cleaner fiber surface during extrusion for the torrefied fiber. During torrefaction the fiber underwent a controlled decomposition in an inert atmosphere, preventing volatiles from being generated, diffusing, and deposited on the fiber surface. When the non-torrefied fiber was introduced to the polymer matrix in the oxygen-rich environment, volatiles created during the uncontrolled decomposition were left on the fiber surface preventing sufficient fiber–matrix interaction, creating a strong odor, and reducing melt flow consistency of the extradite. These volatiles condense during cooling and act much like a plasticizer, enhancing polymer chain movement thus decreasing the overall strength of the composite. These volatiles also more than double the

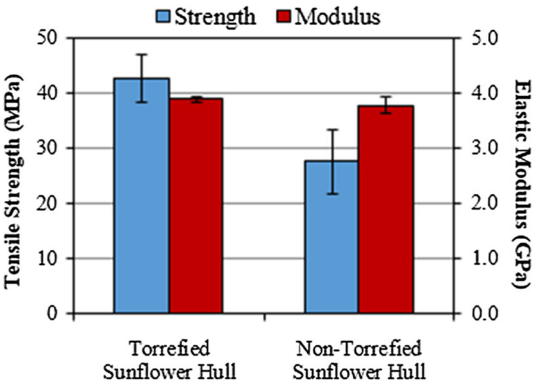


Fig. 1 Tensile comparison between torrefied and untorrefied 20 wt% filled polyamide biocomposites

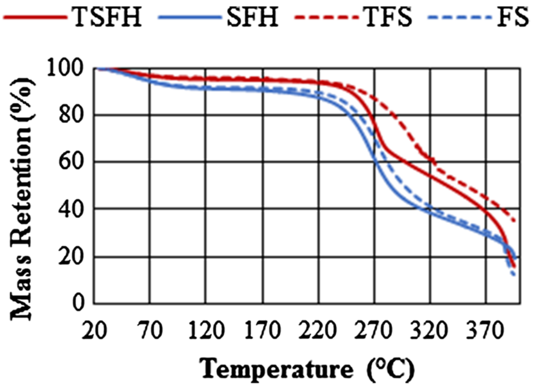


Fig. 3 TGA data of torrefied sunflower hulls, untorrefied sunflower hulls, torrefied flax shive, and untorrefied flax shive

Table 3 Coefficients of variation for torrefied and untorrefied sunflower hull biocomposites

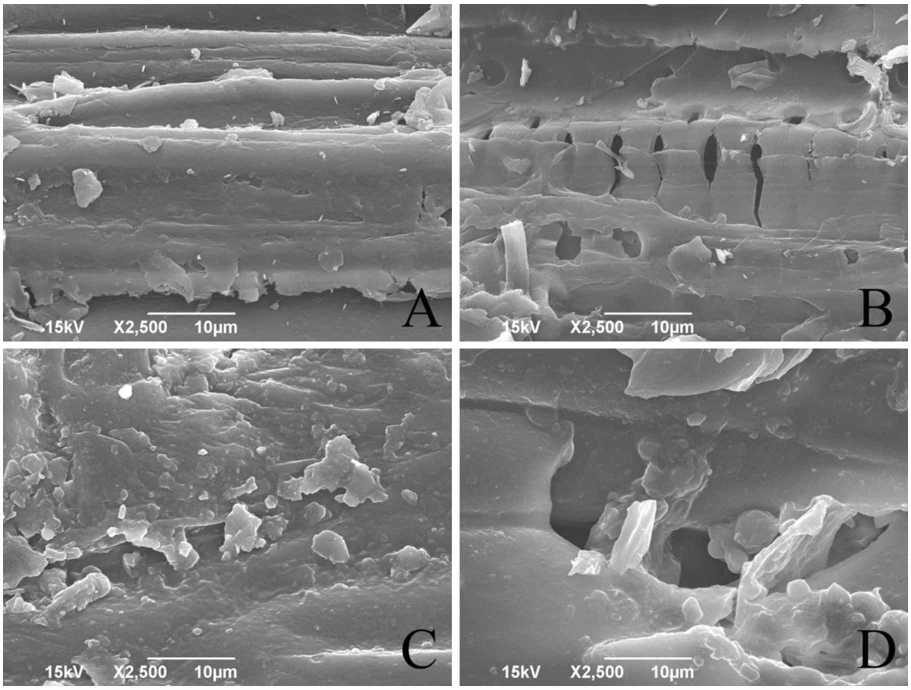
	Tensile strength coefficient of variation (%)	Elastic modulus coefficient of variation (%)
Torrefied sunflower hulls	10.05	0.98
Untorrefied sunflower hulls	20.95	3.73

coefficient of variation among the non-torrefied biocomposites as seen in Table 3.

Figure 2 shows SEM images taken of both torrefied and untorrefied biomass. Images A and C are of the untorrefied flax shive and sunflower hull, respectively. Both fiber surfaces appear to be smooth and free of pores or cracks. Images B and D are of the TFS and TSFH, respectively. The torrefaction process has increased the porosity of the fibers as evident by increased pores and cracks in the torrefied fiber surfaces. The increased porosity of the fibers allow resin to penetrate the fiber surface enhancing surface interaction with the matrix.

Figure 3 shows the increased thermal stability temperature of the torrefied fibers when compared to the untreated fibers through TGA. Torrefaction has increased the 90 wt% thermal stability temperature of sunflower hulls (SFH) from 185 to 249 °C. Similarly, flax shive saw an increase in 90 wt% thermal stability temperature from 215 to 260 °C from torrefaction.

Fig. 2 SEM images of untorrefied flax shive (**a**), torrefied flax shive (**b**), untorrefied sunflower hull (**c**), and torrefied sunflower hull (**d**)



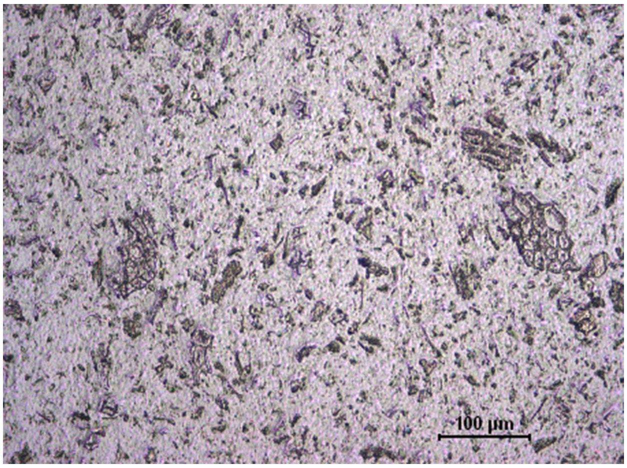


Fig. 4 ×20 optical microscopy image of TSFH biocomposite

Figure 4 shows the fiber distribution of TSFH in PA6 at the 30 wt% loading. This figure shows the even fiber distribution achieved through melt compounding as well as a lack of voids within the specimens. Similar fiber distributions were seen at all loading levels. Figure 5 shows the fiber distribution and voids found in the 30 wt% loading of TFS in PA6. Much like the TSFH the fiber distribution of TFS is even throughout the composite, however, there are voids present in this sample. The voids found in the TFS biocomposites ranged in sizes with the largest voids shown in the figure having a diameter of approximately 300 μm. Similar fiber distributions and void content were seen at all the loading levels of TFS. From the microscopy of both TSFH and TFS biocomposites it can be seen that the filler maintains an aspect ratio of approximately 1–3.

The tensile strength and elastic modulus of the PA6 biocomposites utilizing TFS and TSFH can be seen in Fig. 6. Both the TFS and TSFH produced composites that maintained an average of 70 % of the strength of the neat polymer. Both the TFS and TSFH displayed a slightly decreasing trend in tensile strength with increasing filler loading. The addition of torrefied filler did show an increasing trend in elastic modulus with increasing filler content for both TFS and TSFH. On average the biocomposites increased elastic modulus by 150 % of the neat polymer. During the torrefaction process the hydrophobicity of the natural fiber is increased, changing the surface reactivity and allowing for an enhanced interaction caused by its change in surface polarity between filler and matrix without the need for a compatibilizer. The ability to enhance filler–matrix surface interaction without a costly compatibilizer is promising for traditional polyolefin composites. At the same time torrefaction provides an increased thermal stability allowing for higher processing temperatures before degradation of the filler begins.

Due to equipment limitations only small batches of torrefaction could be conducted for this work, leading to inconsistencies between batches. Each batch produced approximately 120 g of torrefied filler at a yield of 60 % which resulted in the need for three batches of each fiber type to complete the biocomposite test matrix. These inconsistencies resulted in some under torrefied fibers, which can be seen in Fig. 7 as the lighter colored fibers, being introduced into the composite processing. As discussed previously, the under torrefied fibers continued to degrade during the extrusion and injection molding processes acting much like a plasticizer in the final parts. The plasticization effects can be seen in increasing variance and decreased strength with increasing filler loading; as the amount of filler increases so does the effective plasticizer content. If a more consistent torrefaction process can be employed, the variances among final part quality could be reduced and stronger biocomposites could be produced.

The flexural strength and modulus of the TFS and TSFH filled PA6 biocomposites can be seen in Fig. 8. The

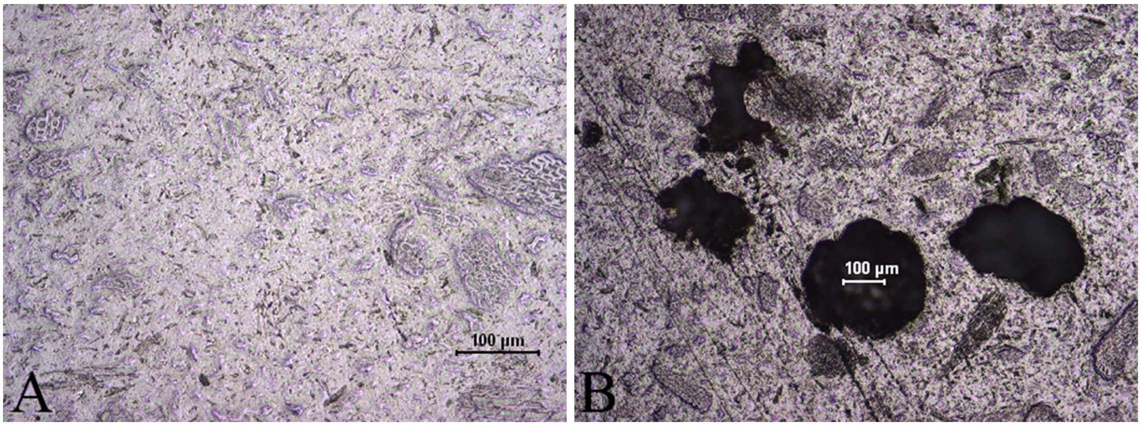


Fig. 5 ×20 optical microscopy image of TFS biocomposite (**a**) and ×10 optical microscopy image of voids present in the TFS biocomposites (**b**)

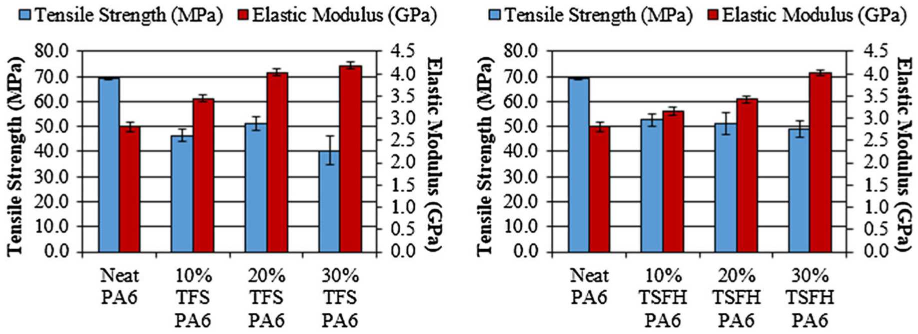


Fig. 6 Tensile properties for torrefied flax shive and torrefied sunflower hull filled polyamide-6 biocomposites

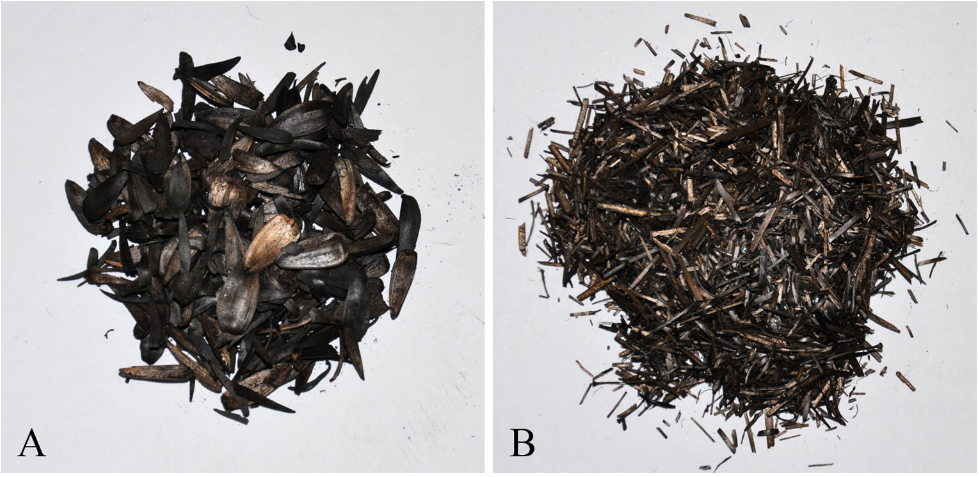


Fig. 7 Photographs of torrefied sunflower hulls (**a**) and torrefied flax shive (**b**)

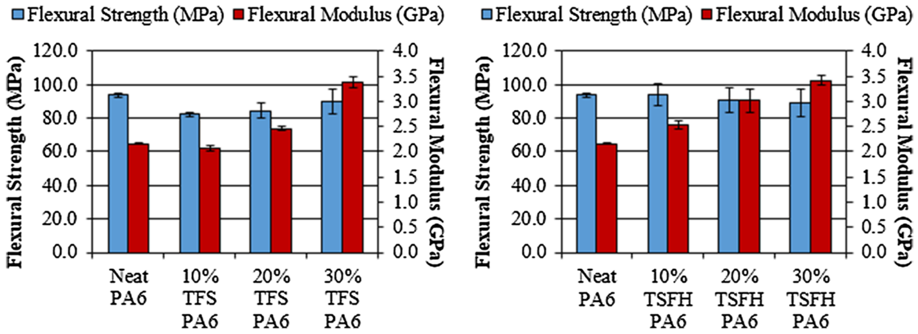


Fig. 8 Flexural properties for torrefied flax shive and torrefied sunflower hull filled polyamide-6 biocomposites

addition of TFS to PA6 showed to maintain on average 94 % of the flexural strength of the neat matrix. This behavior further points to the plasticization effect of un-torrefied fibers. Further investigation using optical microscopy found small micro voids present in the TFS samples, another possible explanation for poor tensile and flexural properties. During the microscopy work no voids were found in the TSFH samples which would indicate the TSFH had a higher degree of torrefaction than the TFS. Both the TFS and TSFH showed an average increase in flexural modulus of 154 % over the neat matrix.

Figure 9 shows the impact toughness of the TFS and TSFH filled PA6. As is expected when adding a filler to a polymer matrix the impact toughness decreases with increasing filler content. As the previous results have indicated some plasticization effects from the under torrefied filler, the impact results from a more consistent and complete torrefaction would look more like the TSFH results. The addition of TSFH decreased the impact toughness by 39 % at the 30 wt% loading, where the decreasing trend appears to have reached a plateau.

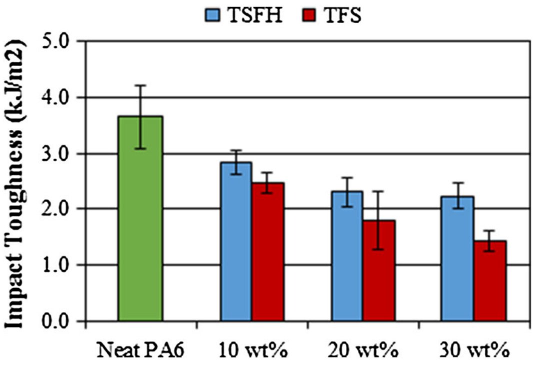


Fig. 9 Impact toughness of torrefied flax shive and torrefied sunflower hull filled polyamide-6 biocomposites

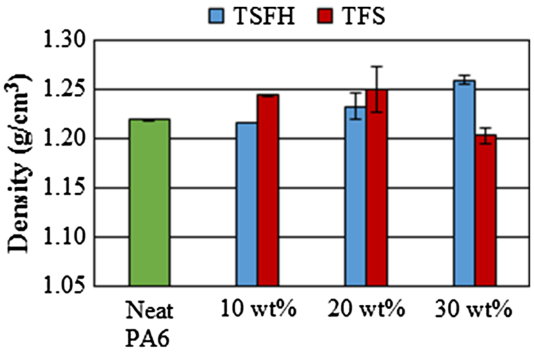


Fig. 11 Immersion densities of torrefied flax shive and torrefied sunflower hull filled polyamide-6 biocomposites

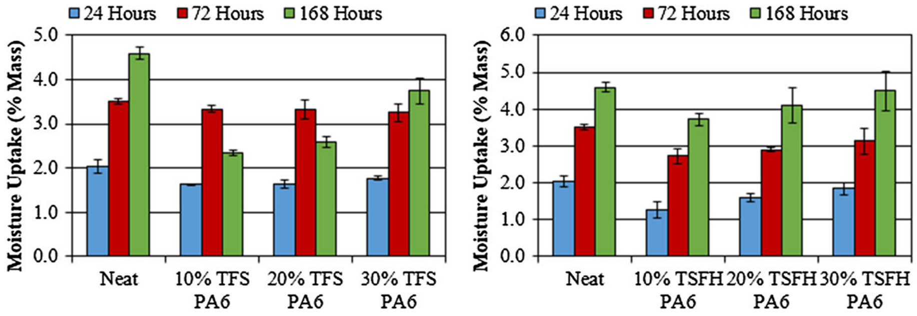
Polyamide materials are known for absorbing moisture form the environment. While the moisture saturation point is around 10 wt% and equilibrium moisture content is approximately 3 wt%, the addition of torrefied biomass has the potential to reduce moisture uptake during end use of the material [21]. The biggest concern with the uptake of moisture for polyamides is the degradation of mechanical performance as the amount of moisture in the material increases.

The left side of Fig. 10 shows the moisture uptake of TFS PA6 biocomposites at 24, 72, and 168 h soaks. All three filler loadings showed to absorb less moisture than the neat matrix at each soak length. However, at the lower filler loadings of TFS the moisture absorption was lower at the 168 h mark than it was at 72 h. To ensure the thermal effects of the moisture analysis did not play a role in the results, new specimens were used for every test in the moisture study. The voids found within the specimens could be filled with absorbed moisture increasing the ultimate moisture content. Another explanation could be that the torrefied filler retards the rate at which moisture is absorbed until the material beings to become saturated. Full saturation was not reached in this study but some future work could shed some light on what is really happening with the TFS biocomposites.

The right side of Fig. 10 shows the moisture uptake results for the TSFH filled PA6 biocomposites. These materials displayed increasing trends with increasing soak times, which is to be expected. The more important trend to note is the decreased moisture uptake with the added filler. All the TSFH filled biocomposites displayed lower moisture uptake compared to the neat matrix. The higher filler loadings did display higher variations than the neat matrix. There are a number of explanations for the variation observed at higher filler loadings; voids within specimens, residuals oils on the fibers acted as a moisture absorption retardant, or the variation within the specimen sizes may have led to induced variation of the moisture absorbed. The TSFH biocomposites did display higher affinity to moisture at the 168 h soak than the TFS biocomposites.

Figure 11 shows the densities of the unfilled PA6 and torrefied biomass biocomposites. The density of the filler was back calculated using the rule-of-mixtures approach. The masses of the filler and matrix were calculated from the dry mass of each sample based on the loading of filler. With no way of verifying the exact loading or void content of each sample it was assumed the loading of each composites was 10, 20, and 30 wt%, respectively, and void free. Based on the given assumptions the density of TFS was calculated to be 1.36 g cm^{-3} with a standard deviation of 0.18 g cm^{-3}. The density of TSFH was calculated to be 1.28 g cm^{-3} with a standard deviation of 0.035 g cm^{-3}. From the optical microscopy images in Fig. 5 the TFS samples do contain voids, taking the void content into account the density of TFS would be higher than calculated. The calculated

Fig. 10 Moisture uptake of torrefied flax shive and torrefied sunflower hull filled polyamide-6 biocomposites at 24 and 72 h soaks



densities of both the TSFH and TFS fall well below the 2.5 g cm^{-3} average density of the traditional glass fiber used in polyamide composites. By replacing fiber glass composites with torrefied biomass biocomposites the overall content of petroleum-based materials is reduced while the final weight of a part is reduced.

Conclusions

Through the torrefaction of flax shive and sunflower hulls, polyamide biocomposites have been successfully produced. By converting the hemicellulose, fats, waxes, et cetera that degrade at a lower temperatures, a more stable biomass filler has been created that can better withstand the increased processing temperatures of PA6. TFS was shown to improve the elastic modulus of PA6 while maintaining similar tensile strengths. Flexural properties were also improved with increased filler loading. As would be expected, the impact toughness of TFS reinforced PA6 saw some decrease from the neat matrix. Voids found within the TFS-filled specimens gave some indication that the torrefaction process was not complete or uniform. TSFH was shown to increase the elastic modulus of the neat PA6, while maintaining a tensile strength similar to that of the neat polymer. Flexural properties were maintained or improved upon the neat matrix, with some loss in impact toughness. The addition of torrefied filler also showed to decrease the moisture uptake when compared to the neat matrix. Torrefied biomass filled biocomposites showed similar mechanical strength to that of the neat polyamide all while reducing the amount of petroleum rich thermoplastic used in the final part which shows to be promising for applications in the automotive industry.

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